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A method of training AFAL/RKPL personnel in the use of the Rheometrics Stress Rheometer (RSR) was needed to reduce the large amount of time required to train an individual to operate the RSR. This paper serves as a guide to the RSR for the newcomer. By working with the laboratory's technicians, I was able to determine what information needed to be provided to the newcomer immediately, and which information could easily be obtained from the RSR's operations guide.

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FINAL REPORT

Rheometrics Stress Rheometer Applications

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Rheometrics Stress Rheometer Applications

by

William Geisler

ABSTRACT

A method of training AFAL-RKPL personnel in the use of the Rheometrics Stress Rheometer (RSR) was needed to reduce the large amount of time required to train an individual to operate the RSR. The report serves as a guide to the RSR for the newcomer. By working with the laboratory's technicians I was able to determine what information needed to be provided to the newcomer immediately and which information could easily be obtained from the RSR's operations guide.

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I. INTRODUCTION:

The method of rheological characterization of highly filled polymer systems varies greatly from manufacturer to manufacturer and even between research groups working with similar materials. Different methods of evaluating highly filled suspensions, and even the testing schedule of a sample can yield rather disparate results from experimenter to experimenter for a single material.

The Solid Propellant Laboratory of the USAF Astronautics Laboratory at Edwards Air Force Base is concerned with the development, preparation and testing of solid propellants. Of particular interest in the testing of propellants' rheological properties is continuity and reproducibility of test measurements. The Propellant Laboratory makes use of two Rheometrics, Inc. apparatus in characterizing propellants -- the mechanical spectrometer and the stress rheometer. The Propellant Laboratory has developed a training and operations manual for the mechanical spectrometer and is in need of a simple and effective way to train personnel in the use of the stress rheometer.

My research interests in rheology have been in the area of characterization of highly filled and polymeric systems through constant stress measurements and variable strain testing. In developing programmed control of a stress rheometer I learned a great deal about the operation, control and testing uses of stress rheometers which has aided me in in this summmer effort.

II. OBJECTIVES OF THE RESEARCH EFFORT:

The goal of my research effort at the AFAL Propellant Laboratory is twofold -- the development of a training and troubleshooting guide for the laboratory's Rheometrics Stress Rheometer (RSR); and the initiation of an NK series solid propellant (R-45M based) characterization study. In order to make full use of the laboratory's equipment it is necessary to have an effective way to bring new personnel up to speed on the operation of equipment so that research does not lag. Of particular concern is the number of duties and frequent rotation of tasks placed on the laboratory's technicians. The difficulty then lies in retrieving meaningful data when several operators are involved with the same study. The report emphasizes the most important and basic operations of the RSR -- leaving detailed description of the device's components operation to Rheometrics' standard operations guide.

III. BACKGROUND:

S.L. Smith's (1985) final report, 'The Rheometrics Mechanical Spectrometer (RMS) Solid Propellant Manual' (AFRPL TR-85-012) provides an excellent starting point for introduction to rheology. The 'Rheometrics Stress Rheometer RSR-8600 Operations Manual' (1987) is the primary source of apparatus and operations specifications, as well as calibration procedures, for the RSR. The RSR is used to examine materials' strain response to a constant or varied applied stress. Several types of fixtures are available to use in testing -- parallel plates, cone and plate, and torsion rectangular test fixtures. The parallel plates and cone and plate are used primarily for testing liquids and suspensions, whereas the torsion rectangular fixtures are used to test solid rectangular specimens.

The report is organized according to the path one takes through average testing procedures. The intent is to present the apparatus to the user in a way that promotes the recognition of recurring themes of operations procedures, as well as highlighting crucial testing concerns. Therefore, this is NOT a step-by-step operations guide, but is intended to be a 'friendly' (?) tour guide to the RSR. Some descriptions of operations may seem to be overly simplified or obvious -- but I have found that they are necessary for those who are not familiar with using personal computer (PC) controlled equipment.

IV. OPERATING CONDITIONS:

Make sure that all equipment has been turned on and warmed up for several hours before beginning. The air compressor supplying the RSR's air bearing should not ever be turned off, unless repairs are necessary. Letting the air run continuously to the air bearing seems to reduce the need for cleaning of the air bearing and is reccommended by Rheometrics, Inc. The compressor tank and air lines should be drained of water daily.

If the RSR will not turn on or powers down during operation check the fuses on the back of both the computer control box and the test station. It is important that any damaged fuses be replaced with the appropriate size of fuse. Also, attempt to determine the source of strain on the equipment. If repairs are beyond your ability, pursue a course of repair with your supervisor.

V. FORMATTING A DISK:

Before running the RSR's programming it will be necessary to format a data storage disk. Insert the DOS disk in drive A (the top one) and the disk to be formatted in drive B. Turn on the switch on the side of the computer, and the switch on the video screen. When you get the A> prompt, type FORMAT B: and then press the ENTER key (NOTE: all commands are followed by striking the ENTER key). Follow the instructions given by the format program.

VI. MAKING A BACKUP DISK:

It is important to make sure that there is always a backup copy of your computer programming. The RSR's programming is contained on a disk labeled 'Recap II RSR Composite Disk v 1.31'. If you look through the boxes of disks in the lab you should be able to find a backup copy of this programming. If there ever comes a time when the RSR disk goes bad and won't run the programming, it will be necessary to make a copy of the backup disk. To do this, boot up the DOS disk, (described in V) type DISKCOPY A: B:. Insert the 'Recap II RSR Composite Disk v 1.31' backup disk (the SOURCE disk) in drive A, and a blank formatted disk in drive B (the TARGET disk). The DISKCOPY program will instruct you in copying the backup copy of the RSR's composite disk. Properly label the newly copied disk.

VII. SUBMENU USE (Setting up a Notebook):

Place the 'Recap II RSR Composite Disk v 1.31' in drive A. To reboot the system with this diskette, depress the CTRL, ALT, and DEL keys simultaneously. The IBM will ask you for the date and time. The current time must be typed in in military format. After inputting the date and time the Rheometrics logo is printed on the screen, quickly followed by the main menu.

The Recap program makes use of menus and submenus to control experiments, print and plot data, analyze data and the like. Before running an experiment it will be useful to see how the menus work and practice using them. Submenus may be selected in one of three ways: using the appropriate function keys (F1, F2, F3, etc.) located on the top row of the keyboard; by highlighting the submenus with the up or down arrow keys followed by pressing the ENTER key; OR by pressing a key which corresponds to the appropriate yellow shaded letter.

RSR experiments are stored in notebooks on the data diskette. The notebooks allow you to segregate experiments by project -- each project's experiments will be stored in its own notebook. So, our first order of business will be to setup a notebook for storing future experiments. Press F1 to 'Select Notebook'. A list of notebooks will be presented -- you will note at the bottom of the screen that pressing 'C' is used to create a notebook. So, go ahead and press 'C'. Type in your notebooks name -- try to be descriptive so that you will be able to tell what is contained in the notebook three years hence.

Practice using the three methods of selecting submenus to see what they contain and formulate a mental picture of the organization of the Recap II program. If you find yourself in a submenu with no apparent way out (e.g. when entering 'Terminal Mode') simply press F10 -- this action returns you to the previous menu.

VIII. CHECKING UTILITIES SETTINGS:

From the main menu select F7 -- 'Utilities'. Highlight the option, 'Change the default file settings' and select it by pressing ENTER. The important note to make here is that the Engineering units are setup in SI units. It seems that if cgs units are selected the program will compute the data in cgs units but label the data in SI units! So, be careful when using cgs units.

If your data disk ever becomes full or you need to use another disk make sure that you read the index off of the disk. To do this, select 'Change data disk and read the new index' from the Utilities submenu.

The Utilities submenu contains other useful options. Familiarize yourself with them by looking through the submenu.

IX. CALIBRATING THERMOCOUPLES:

Thermocouples and the PRT (Platinum Resistance Thermocouple) should be calibrated periodically. The PRT is used by the computer's temperature controller to meet the setpoint dialed in on the TEMP thumbwheel when the oven is activated. The Rheometrics RSR Operations manual gives a well organized breakdown of calibration in section 6-3.

X. TEMPERATURE CONTROL:

Temperature control is fairly simple when using the RSR. If you wish to run a test at a temperature lower than room temperature, liquid nitrogen is used. If an elevated temperature is desired, air heating is used.

Before heating or cooling the chamber the appropriate thermocouple should be selected. Tests involving parallel plates or the cone and plate fixture should make use of the thermocouple located underneath the bottom fixture.

Rectangular torsion tests use the thermocouple just to the right of the lower fixture. The thermocouples are plugged into the test station on the front left panel.

XI. COOLING:

1. Have your fellow technicians or supervisor check you out on the filling and operation of the indoor nitrogen storage tank. The inside tank is filled from the large outdoor nitrogen reservoir. The vapor pressure in the outdoor tank should not be allowed to get so great (50-60 psig) that the tank's blow off valve is activated.
2. Open the liquid valve on the indoor tank. Make sure that the valve is not fully opened -- as the valve might stick in the open position. When a valve has been fully opened turn it back 1/4 turn. Open the gate valve on the line leading to the RSR's small nitrogen dewar.
3. Close the doors of the oven -- enclosing the upper and lower fixtures. The oven will not operate with the oven doors open. Depress the 'LN2/GAS' switch on the environmental control box so that 'LN2' lights. Set the temperature thumbwheel to a higher temperature than will actually be used. Press the oven 'ON' button on the RSR test station, wait a few minutes, and then dial in your desired temperature. By doing this you have avoided a rapid temperature dive.
4. Wait for the lines and dewar to fill with liquid nitrogen (5-10 minutes). When this has been accomplished

the 'LN2 READY' light will come on. If this procedure takes an excessive amount of time, check the inside tank pressure and level, and the liquid nitrogen lines for leaks.

XII. HEATING:

1. Make sure liquid nitrogen line valves are closed.
2. Put the 'LN2/GAS' switch in the 'GAS' position.
3. Lower the upper fixture so that it will fit in the oven.
4. Shut the oven door. Set the temperature thumbwheel at a setpoint lower than the desired temperatures.
5. Press the oven 'ON' switch, wait a few minutes and dial in the desired temperature.

XIII. MAKING A 'CAL. CURVE':

Basically, a Cal. Curve is a plot of the resistance of the air bearing versus position. While running a test the RSR compensates for error in readings by accounting for friction losses and non-uniform laminar flow in the air bearing. Cal. Curves may be used for experiments 50 C above or below the temperature at which a Cal. Curve was measured. For example, a Cal. Curve measured at 100 C may be used when testing a material at 120 C. Naturally, if you are making very sensitive readings it may be necessary to make Cal. Curve measurements at the temperature of your test. It must be kept in mind that Cal. Curves are fixture dependent -- you shouldn't use a Cal. Curve made with the 50 mm parallel plates to run a test with a cone and plate. For normal operating conditions, Cal. Curves should be remeasured every few months.

To make a Cal. Curve, lower the upper fixture until it will fit inside the oven. Close the oven doors. Bring the oven to temperature for 20-30 minutes. Setup a notebook for storing your Cal. Curves. Press the 'TRACK' button and wait for the 'POSITION LOCKED' light to come on. Press 'CAL'. The computer will take its torque measurements during the following 15-20 minutes. When the 'CAL' light turns off the Cal. Curve test is finished. The Cal. Curve needs to be stored on your data disk. From the main menu select 'CALIBRATION' (F8). Store the Cal. Curve in the 'CALIBRATION' submenu by following the directions given by the 'Store Cal. Curve' (F1) option. Note that Cal. Curves are retrieved and deleted through the CALIBRATION submenu. When loading a Cal. Curve remember to select your calibration notebook before retrieving a Cal. Curve. It is important to check which Cal. Curve has been loaded into the computer before beginning an experiment.

XIV. PLATE GAP:

Setting the zero plate gap takes a bit of practice. The zero gap is the position of the fixtures when they initially meet. The zero gap should be set before each experiment. If the zero gap is set incorrectly, experimental results are thrown off -- so you should recheck the zero gap several times before testing a material.

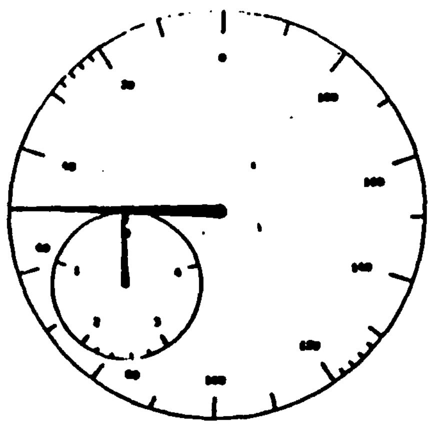
The quill is lowered and raised with the hand crank located on the top of the test station. Lower the quill -- as the top fixture gets close to the bottom, watch the 'ZERO GAP INDICATOR' on the test station carefully. Lower the upper fixture a few microns at a time. When the needle on the gap indicator deflects the plates have come into contact. Adjust the quill height until you find the position where the plates just meet. To set the gap gauge to zero, adjust the thumbscrew under the gauge so that both needles read zero. Raise the quill and recheck your zero gap several times.

Gap readings are made in millimeters. On the RSR gap gauge there are two needles to be read when determining the gap height. The small gauge in the middle is read first and is read in millimeters -- the small tick marks on this gauge each represent 0.2 mm. The gauge with the large needle is read next. Each small tick mark on the larger gauge represents 0.002 mm (2 microns). One trip around the larger gauge represents 0.2 mm. A numbered mark like 40 on the larger gauge is read as 0.040 mm (40 microns). Figure 1 shows some examples of gauge settings.

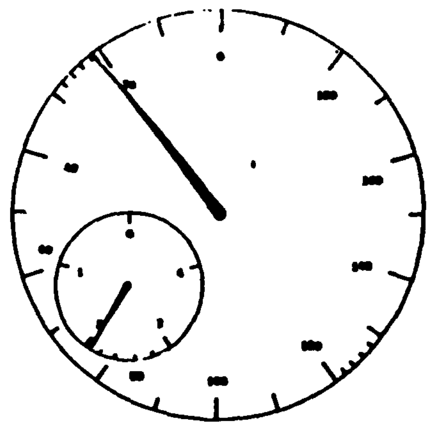
XV. TORQUE CALIBRATION:

Due to the organization of this guide it will be helpful to try some practice experiments (see XVI) before attempting to calibrate the RSR. However, I feel it is important to be presented with the torque calibration

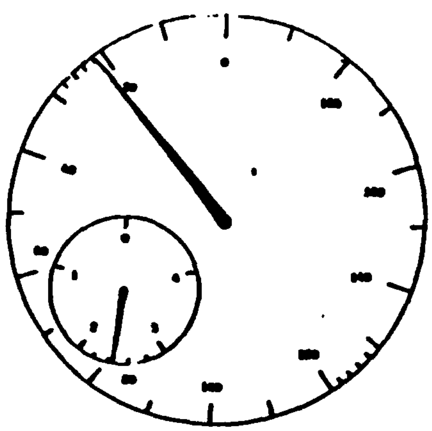
FIGURE 1 - Example readings of RSR gap gauge.



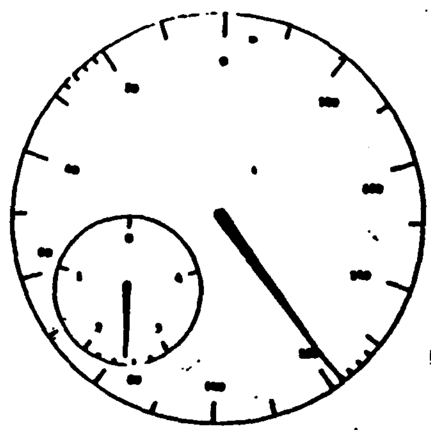
0.050 mm (50 microns)



2.022 mm



2.422 mm



2.522 mm

procedure before you begin to perform experiments. Hopefully, after reading this section you will remember that the RSR will need to be calibrated periodically.

Rheometrics' operations manual gives an easy to follow breakdown of the calibration procedure in section 6-1.2. It is important to go through this procedure several times (at least three) before making a decision on whether or not to calibrate the RSR. Plate gap and temperature control should be carefully set during this procedure. You should not expect to match the viscosity given on the oil standard bottle exactly. If your viscosity measurements are within 5% of the value given by the manufacturer you are doing great and the RSR isn't in need of calibration. If the RSR's results are consistently more than 10% high or low you will need to continue with the calibration procedure. Again, make sure that you make several measurements of the oil's viscosity, and that you get consistent results before you calibrate the torque. If you are not sure of the age of your oil standard or the oil's purity, get a new standard oil.

XVI. STRESS MODE EXPERIMENTS:

Through stress mode experiments the operator may run constant stress tests. In this section I will demonstrate the use of the 50 mm parallel plates. The use of the other fixtures is similar and a small amount of instruction is given for the cone and plate and torsion rectangular fixtures in the latter part of the section.

When running a stress mode experiment you will often have to determine the stress level input that will yield reasonable results. The RSR's experimental limiter is rotational rate. If a fixture is turning too slowly the RSR's transducers cannot pickup any movement. Rate limitations are listed in Tables 4-1 and 4-2 of the RSR's operations manual.

I will use a propellant to demonstrate the use of the parallel plates. NK-33A is an 85% solid filled propellant. When testing solid propellants it is desirable to test them immediately following mixing. Nk-33A was mixed at about 60 C -- so I stabilized the oven temperature at 60 C prior to the end of the mix (see XII). While waiting for the mix I checked and rechecked the plate gap (see XIV). Next, I set the 'TEST GEOMETRY' and 'TEST PARAMETERS':

1. From the main menu select 'Terminal Mode' (F6).
2. Press the 'TEST GEOMETRY' button on the computer control box.
3. Enter 'Y' for 'DISC & PLATE' and 'N' for the other plate choices.
4. I will set the 'GAP' for my test at 2.000 mm. The plates I am using have a 25 mm radius.

If you make a mistake while entering information there are two ways to correct the misinformation -- begin the process over, or while you are on the line where the error has occurred, press the CTRL and K keys simultaneously.

5. Press the 'TEST PARAMETERS' button.

6. Answer the 'USE EXTERNAL INPUT?' question with 'N'.

7. Answer 'Y' to the 'STRESS MODE' question.

The RSR has 8 command zones -- ZONES 1-4 are designated as stress zones, while 5-8 are recovery zones. The user inputted stress is applied to the test material during stress zones. During a recovery zone, no stress is applied and the RSR measures movement of the upper fixture caused by the material's recovery. The total length of time an experiment may run can not exceed 32,760 seconds. If you exceed this time limit, you will not be able to analyze the data taken during the test.

8. I entered 50 dynes/sq. cm as a stress, for 600 seconds in ZONE 1. Zeroes were entered for the other zones.

Before loading the sample, press the 'TRACK' button. When the 'POSITION LOCKED' light comes on, raise the fixture and load your sample. Press 'START' to begin testing.

When the experiment ends, return to the main menu (F10). To store the experiment, select 'Store Experiment on Disk' (F3). The program will ask you to title your experiment and provide a space to type in any observations you made about the test. After typing your notes, press the ESC key to continue with the data storage procedure.

Printing and plotting menus are also selected from the main menu (F5). Before printing or plotting data, run through the printing or plotting parameters submenus (F3, F4). Your answers will vary according to the type of test being performed. If you have not used a printer or plotter before, ask one of your fellow technicians to explain the use of the printer and/or plotter. The 'Printing Parameters Selection' (F3) option is fairly straightforward -- simply answer each of the questions the program poses. When filling in t 'Axes Range Selection' portion of the 'Plotting Parameters Selection' (F4) option, you will find the high and low data points listed in the 'Actual Window Value' box. To turn on or off the ability to send information to the printer or plotter press Alt F2 or Alt F3 respectfully.

A straight line, least squares fit can be made to any range of data by selecting 'Least Square Fit Analysis' (F5). Viscosity measurements are determined by using option (F1) 'Stress Zone'. Use the data at the end of a stress zone which is fairly linear to determine a steady state viscosity.

Cone and plate experiments are performed in an analogous fashion to the parallel plate experiments. A few methods are different however -- the gap of the 25 mm, 0.1 radian cone angle fixtures are set at 0.050 mm (50 microns); and tests must be performed at constant temperatures to maintain the appropriate gap.

The torsion rectangular fixtures are used to study rectangular solid samples. The thermocouple located underneath the bottom fixture will need to be removed temporarily during experimentation. The dimension of samples are measured with calipers and are repoted in terms of millimeters.

XVII. STRESS RAMP EXPERIMENTS:

Stress ramp experiments are selected from the 'Terminal Mode' (F3) with the 'TEST PARAMETERS' button. Simply answer 'N' to the 'STRESS MODE' question and 'Y' to the 'STRESS RAMP' option. You will be asked for the maximum applied stress and the duration of the experiment. When the test is run, a linear ramp of stress will be inputted over the duration of the experiment. The stress ramp test may be used to determine a materials yield stress.

XVIII. RECOMMENDATIONS:

Due to the short visit I was afforded to the propellant laboratory, several areas of this report are slightly less than I had hoped they would be. In particular it would have been nice to work through some actual experiments in the body of the report. However, time and the twenty page format limited the inclusion of detailed example experiments. Due to the nature of vacation seasons, I was unsuccessful in scheduling propellant mixes with the mixing laboratory during my five week stay at AFAL-RKPL.

However, I do feel that the report will be a useful starting point in the training of personnel on the use of the RSR. I have stressed the need for cautious and mindful experimentation.

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REFERENCES

Rheometrics, Inc., 'Rheometrics Stress Rheometer RSR-8600 Operations Manual', 1987.

Smith, S.L., 'The Rheometrics Mechanical Spectrometer (RMS) Solid Propellant Manual', AFRPL TR-85-012, 1985.